The BESSY Fluorescence Chamber

1 Overview

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Typical Experiments

X-ray absorption experiments (EXAFS, NEXAFS etc.)

General Informations

The fluorescence chamber is a comparatively simple unit for x-ray absorption experiments on solids, surfaces and interfaces with a semiconductor x-ray detector. The ultra-high vacuum chamber is evacuated by a turbomolecular pump (240 l/s) and a titanium sublimation pump. The base pressure is typically $5 \cdot 10^{-9}$ *mbar*.

X-ray Detector

A high purity germanium detector is fixed 90° with respect to the incident synchrotron radiation. Depending on the space at the used beamline, it also can be used fixed with an angle of 45°. The working distance between sample and detector can be varied from 5 *mm* to 140 *mm*. The best energy resolution is 85 *eV* at 290 *eV* (C 1s) and 110 *eV* at 1740 *eV* (Si 1s). In particular, the detector is suited for studies of bulk materials and buried layers.

I_0 Detector

A gold grid (80% transmission) controls the incident synchrotron radiation by the generated photo electrons. I_0 is used for the normalization of the measured absorption data.

Electron Yield Detector

With the electron yield detector total yield measurements in x-ray absorption experiments are possible. It is fixed 45° with respect to the incident synchrotron radiation. The working distance is 100 *mm*.

Manipulator

The manipulator allows movements in the following regions: x, y ±15 *mm*; z: 300 *mm*; Θ: 0...360°. As well for room temperature as for cooled measurements (liquid nitrogen) there is a sample port where samples can be placed under UHV conditions using a sample transfer mechanism (see below).

Sample Preparation

If required, a sputter gun (5..10 kV Ar-ions) can be used inside the chamber. Otherwise the samples can be prepared in the BESSY chemical lab.

Computer Control

Hardware:	IBM-AT compatible computer (386)
	PCA-II interface card for the germanium detector
Software:	PCA-II program for pulse heights analysis

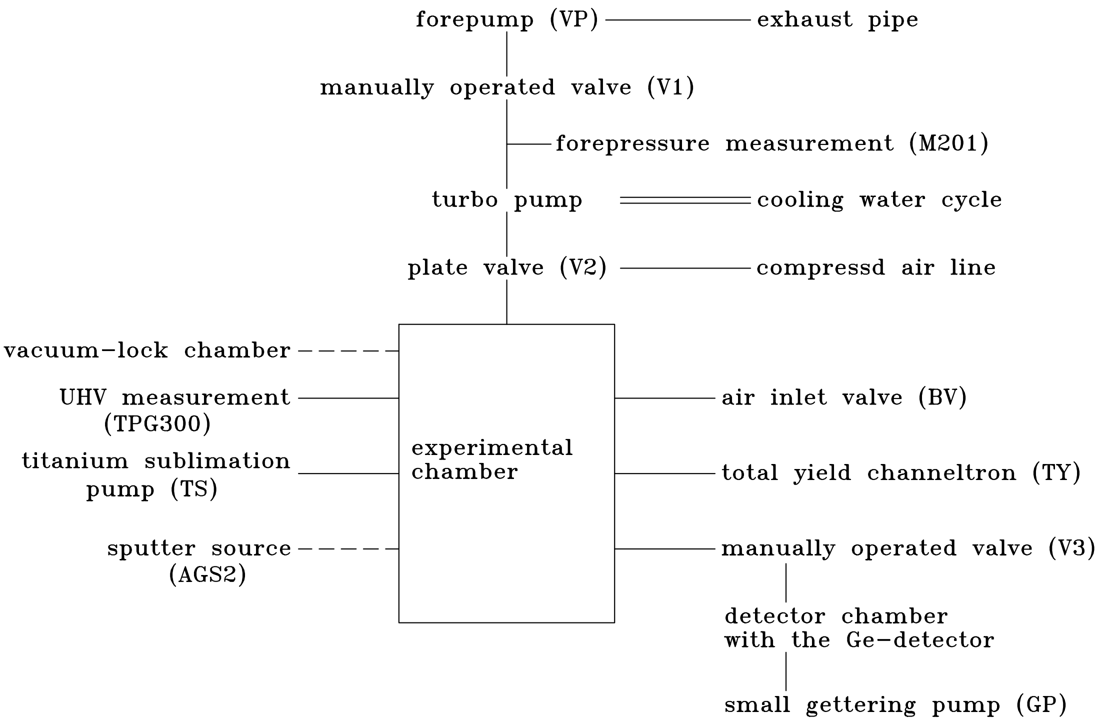
General Notes about Working at Experimental Stations

You should keep the experimental station (measurement chamber, beam line and equipment) clean and in order - also in your own interest. In addition, you should remember everytime the safety regulations (especially those about radiation safety and vacuum).

2 Pumping Down and Venting the Fluorescence Chamber

Pumping Down

1. Connect all vacuum parts:



2. Connect media (compressed air, cooling water, exhaust gas line, power).
3. Close valves BV, V1 and the valve to the air-lock. Open valve V2.
4. Switch on pumping unit (forepump VP and turbo pump TP) with the right button on the controller.
5. Open manually operated valve V1. Switch on measuring instrument M201.

6. The turbo pump will reach step 2 after $t \approx 5$ *min* and step 6 after $t \approx 13$ *min*.

7. Turn on UHV measuring gauge TPG300 below $p < 2 \times 10^{-5}$ *mbar*.

Venting

1. Close valve to the beam line and to the air-lock.

2. Move Ge detector totally out of the experimental chamber. Close manually operated valve V3.

3. Disconnect total yield detector (battery box, NIM chassis).

4. Turn off I_0 measuring instrument.

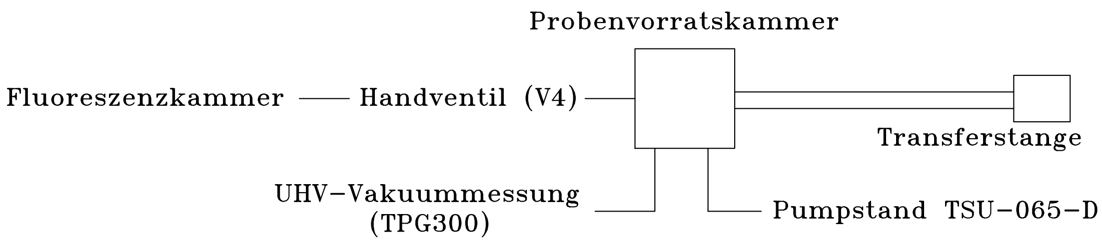
5. Venting with liquid nitrogen through the venting valve (BV):

   - Open BV slowly until $p_{TPG300} = 10^{-4}$ *mbar*.
   - Close manually operated valve V1.
   - Switch off pumping unit.
   - Turn off UHV measuring instrument TPG300.
   - Open BV more. If TP makes too much noise while speeding down close BV a little bit.
   - Open BV totally after the turbo pump has come to a standstill.
   - Turn off forepressure measuring instrument M201.

6. The chamber is vented when the manipulator bellow has become loose.

3 Using the Air-Lock

The air-lock is generally attached to the fluorescence chamber. It is connected through th manually operated valve V4 with the fluorescence chamber. The following sketch shows the setup.



Venting the Air-Lock

1. The valve to the fluorescence chamber must be closed.

2. Liquid nitrogen is used for venting.

3. Switvh off UHV measuring gauge (TPG300).

4. The automatic venting valve at the turbo pump is used for venting.

5. Switch off the pumping unit TSU-065-D. The air-lock vents automatically.

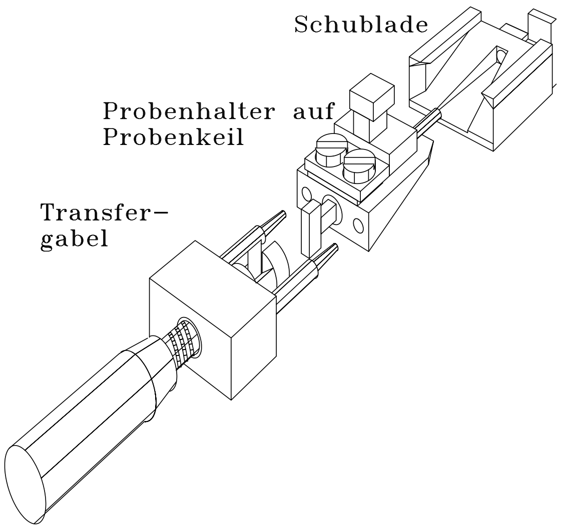
Now the air-lock can be opened at the z-translator. Then you can take off the rotary drive with the sample storage. The samples can be removed and replaced.

Pumping Down the Air-Lock

1. Switch on pumping unit TSU-065-D. The turbo pump will reach normal operation after 3 to 5 min.
2. Switch on UHV measuring gauge (TPG300).
3. The valve V4 can be opened if the difference between the pressure in the fluorescence chamber and the pressure in the air-lock is a factor of 10. The pressure in the fluorescence chamber will then double after opening the valve V4.

Transfering Samples

The components of the transfer system are shown in the following sketch. To transfer a sample, thread the transfer fork heads into the bore holes of the wedge of the sample holder. Push the transfer head - maybe with light



turning of it - as far as the screw of the sample holder is in the locking position of the transfer head. Then unscrew the sample holder from the sample port by turning counterclockwise. The screw of the sample holder must be locked! Then the sample holder can be pulled out of the sample port.

For screwing the sample holder into the sample port push it straight into th port. By turning it clockwise it will be screwed into the port and be unlocked. Then the transfer head can be pulled back.

IMPORTANT:
Do not transfer if the sample holder is not locked in the transfer head.
Do not close the manually operated valve V4 (between air-lock and fluorescence chamber) if the transfer rod has not pulled back completely.
Do not screw the sample with force.

4 The Germanium Fluorescence Detector

Normal pressure:
$5 \cdot 10^{-9}$ *mbar* in the detector chamber (= 2 μA at the small gettering pump)
Detector cooling:
Use of liquid nitrogen (7 *l* dewar). **One filling will be enough for six days, that means you should refill it twice a week**. Without cooling the small gettering pump can't maintain the vacuum. A half-full dewar leads to a N_2 alarm (accoustic as well as visual) at the TX1236 module if the detector electronics are turned on.

Moving into Measuring Postition

1. Close valve to beam line.
2. Turn the handwheel of the valve between detector and experimental chamber V3 until the red pin juts out completely (≈ 5 *mm*) from the handwheel.
3. Move the detector by constant and counterclockwise rotation of the handle into the experimental chamber.
4. Mark the position of the smallest distance between detector and sample manipulator (observed through a viewport). The bellow can be compressed down to 8 *cm*.
5. Darken the experimental chamber, turn off all light sources (filaments of measuring gauges, titanium sublimation pump, ...).
6. Turn on the detector electronics (NIM chassis).

Finishing the Measurement

1. Close valve to beam line.
2. Turn off the detector electronics (NIM chassis).
3. Move the detector out of the chamber constantly by clockwise rotation of the handle (arrow direction $\rightarrow$) to the stop.
4. Close the valve between detector and experimental chamber by a constant rotation of the handwheel until the red pin disappears totally in the handwheel and you feel a resistance in the rotation.

Detector Electronics

If light (zero order, filaments of measuring gauges, titanium sublimation pump, lighting of the sample area for adjustments, ...) falls into the experimental chamber the detector electronics must be turned off!

1. Turn on the PC. Start program `pca.bat` or `pcaklm.bat`.
2. <u>Electronic connections</u>
Detector cable with pulse processorr TX1255 (in NIM chassis)
Pulse processor with voltage module TX1236 (in NIM chassis)
Pulse processor FD with rate meter INPUT (in NIM chassis)
Pulse processor INPUT with TC245 Signal-IN (in NIM chassis)
Pulse processor OUTPUT or TC245 UNI-OUT with PCA interface card INPUT (in PC, right).
3. Turn on NIM chassis.
4. After 30 *min* the initialization of the electronic should be finished and the detector can be used for measurements.
Dark count rates at the TX1255:
   - high count rate: 100 cps ($\rightarrow$ rate meter), correction with fast disk.
   - low count rate: 200 cps, correction with slow disk.

Standard Amplifier Adjustments

Pulse processor TX1255:	Gain 4
Tennelec TC245:	Gain grob 500, fein 13
	PUR - OFF
	PA-RESET

Ranges of linear amplification
(Measure the total count rate at the rate meter!)

- Pulse processor TX1255
  (no shifts of the energy calibration)
  low count rate : up to 2500 cps
  medium count rate: up to 5000 cps
  high count rate : up to 7000 cps
- TC 245 (peaking time)
  (shifts of the energy calibration coresponding to peaking time.)
  24 μsec: up to 3000 cps
  12 μsec: up to 7000 cps
  3 μsec: up to 20000 cps

The intensity can be varied by the distance between detector and sample.

PCA-II Interface Card

The PCA interface card allows pulse heights analysis of the peaks formed by the amplifiers, graphic representation, energy calibration, choice of an interesting spectral range (setting a window) and delivery of TTL signals at the outputs. It's possible to save pulse heights spectra either in ASCII or binary.

- Standard adjustments:

MODE	PHA (pulse heights analysis)
Memory Group	E1 (x-axis: 1024 memory blocks)
Conversion Gain	2048 (10 V INPUT = memory 2048)
Display	1024 (x-axis: 1024 pixel)

- Setting a window and outputs:
  1. SCA
     - Acceptance range of the interface card
     - Adjustment with a rotatory resistor (U, L on the back side of the interface card)
  2. LTB
     - Choose ROI (region of interest) by software (F9, F10, Del, Alt-R)
- Important keys:

F1	Start Measurement (on) - Finish Measurement (off)
F2	Disable erasing - enable erasing
Strg-F2	Erase spectra
Alt-..	Activate menu (.. = 1. letter)
←,→	Move cursor on x-axis (=energy).
↑,↓	Scale y-axis (linear .. logarithmic)
right mouse button	Turn from menu to cursor
left mouse button	Execute operation

Further information and help in the PCA-II manual

Detector De-icing

Because of the permanent detector cooling a thin ice layer grows on the detector. The ice layer causes a stronger absorption of the x-ray radiation. While analysing energies above the oxygen K edge one will have additional O-K_α radiation which will lead to further errors in intensity (high O peak at 525 eV, stronger absorption of other lines). Therefor the detector should be de-iced before each measurement period. For this reason a de-icing control unit is used which heats the head of the detector cyclically and regulatedly. The pressure increases for a short time up to 10^{-5} $mbar$. This forbids to do this procedure inside the detector chamber.

1. Move the detector into the experimental chamber (The valve to the beamline must be closed).
2. Unplug the LN (liquid nitrogen) sensor from the pre-amp housing (LN alarm at module TX1236 activated!).
3. Plug in the detector de-icer control box into the LN sensor jack located on the pre-amp housing.
4. The green "COMPLETE" led should be lit. This indicates there is power to the control box.
5. Push the button on the detector de-icer control box front panel. The "COMPLETE" led should go off and the red "DE-ICING" led should light.
6. The "DE-ICING" led will remain on for approximately 1 hour. When the "COMPLETE" led comes on the detector should be de-iced.
7. Unplug the detector de-icer control box from the pre-amp housing and plug in the LN sensor.
8. The detector can now be used but it may take a while for the detector to completely settle down ($\approx 30 min$).

5 Total Yield Measurements

Connections: